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Nanotechnologies — Characterization of single-wall carbon nanotubes using ultraviolet-visible-near infrared (UV-Vis-NIR) absorption spectroscopy

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National foreword

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Nanotechnologies — Characterization of single-wall carbon nanotubes using ultraviolet-visible-near infrared (UV-Vis-NIR) absorption spectroscopy

Nanotechnologies — Caractérisation des nanotubes à simple couche de carbone par utilisation de la spectroscopie d'absorption UV-Vis-NIR

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Foreword

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This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

This second edition cancels and replaces the first edition (ISO/TS 10868:2011), which has been technically revised.

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Nanotechnologies — Characterization of single-wall carbon nanotubes using ultraviolet-visible-near infrared (UV-Vis-NIR) absorption spectroscopy

1 Scope

This document provides guidelines for the characterization of compounds containing single-wall carbon nanotubes (SWCNTs) by using optical absorption spectroscopy.

The aim of this document is to describe a measurement method to characterize the diameter, the purity, and the ratio of metallic SWCNTs to the total SWCNT content in the sample.

The analysis of the nanotube diameter is applicable for the diameter range from 1 nm to 2 nm.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/TS 80004-4, *Nanotechnologies — Vocabulary — Part 4: Nanostructured materials*

3 Terms, definitions and abbreviated terms

For the purposes of this document, the terms and definitions given in ISO/TS 80004-4 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1 Terms and definitions

3.1.1
purity indicator
optically defined indicator of the ratio of the mass fraction of SWCNTs to the total carbonaceous content in a sample

Note 1 to entry: Purity indicator is NOT "purity" itself which is defined as the percentage of mass of SWCNTs to the total mass of the sample. This guideline cannot evaluate this general purity because absorption spectroscopy cannot detect metallic impurities that are generally contained in any SWCNT sample. In order to characterize metal impurity content, there is a different Technical Specification on thermogravimetric analysis. Metallic impurity is defined as catalytic metal particle and does not include metallic carbon nanotube. See ISO TS 11308.

3.1.2
ratio of metallic SWCNTs
optically defined compositional ratio of metallic SWCNTs to the total SWCNTs contained in the sample

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3.2 Abbreviated terms

For the purposes of this document, the following abbreviated terms apply.

CMC	Sodium carboxymethylcellulose
DMF	Dimethylformamide
DOS	Density of states
NIR	Near infrared
NMP	N-Methyl-2-Pyrrolidone
SC	Sodium cholate
DOC	Sodium Deoxycholate
SDS	Sodium dodecyl sulfate
SDBS	Sodium dodecylbenzene sulfonate
SWCNT	Single-wall carbon nanotube
TEM	Transmission electron microscope
UV	Ultraviolet
VHS	van Hove singularity
Vis	Visible

4 Principle

4.1 General

All SWCNT samples contain both semiconducting and metallic SWCNTs, together with impurities consisting of carbon and other elements unless the samples have been altered after production. UV-Vis-NIR absorption spectroscopy can be used for the measurement of interband optical transitions specific to SWCNTs. The analysis of these optical transitions provides qualitative and semiquantitative information important for the characterization of SWCNT samples, such as mean diameter, purity, and the ratio of metallic SWCNTs to the total SWCNT content.

4.2 UV-Vis-NIR absorption spectroscopy

The intensity of light passing at a specified wavelength, λ, through a specimen (I) is measured and it is compared to the intensity of light before it passes through the specimen (I_0). The ratio I/I_0 is called a transmittance. The absorbance, A, is expressed as $-\log(I/I_0)$. The plot of the absorbance against wavelength for a particular compound is referred to as an absorption spectrum.

NOTE The relationship between transmittance and absorbance is only rigorously correct when reflectance is negligible and there is no scattering.

4.3 Optical absorption peaks of SWCNTs in the UV-Vis-NIR region

The shape of the electronic DOS of semiconducting and metallic SWCNTs shown in Figure 1 is a series of spikes that are referred to as VHS. The peaks observed in the optical absorption spectra of SWCNTs are attributed to the electronic transitions between these VHSs as shown by arrows in Figure 1. S_{11} and S_{22} are used as the symbols of the absorption due to the first and second interband transitions of

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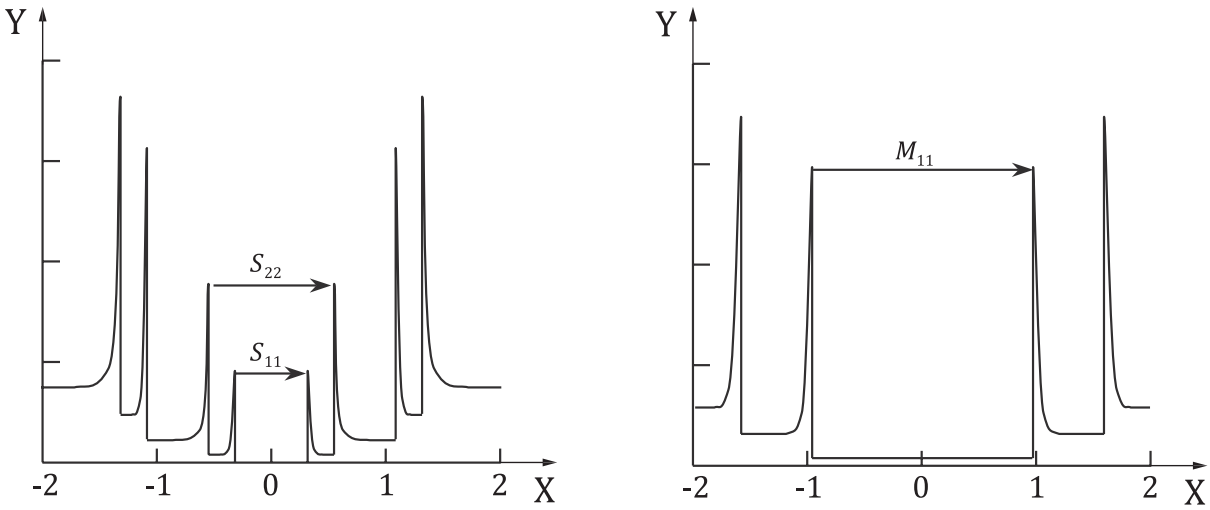
semiconducting SWCNTs, respectively [see Figure 1 a)]. M_{11} means the absorption arising from the first interband transition of metallic SWCNTs [see Figure 1 b)].



a) Electronic DOS of semiconducting SWCNTs **b) Electronic DOS of metallic SWCNTs**

Key

X energy (eV)

Y electronic DOS (arbitrary unit)

S_{11} first interband optical transition attributed to semiconducting SWCNTs

S_{22} second interband optical transition attributed to semiconducting SWCNTs

M_{11} first interband optical transition attributed to metallic SWCNTs

NOTE 1 Arrows represent interband transitions that result in optical absorption.

NOTE 2 See Reference [2].

Figure 1 — Electronic DOS diagram of SWCNTs near the Fermi level

To interpret the absorption spectra of SWCNTs, band structures calculated using the zone-folding method are frequently used. The electronic structure of an SWCNT is generally given by that of a two-dimensional graphite sheet expressed by the tight binding approximation as shown in Formula (1)[2]:

$$E_{2D} = \pm\ \gamma \left[1 \pm 4\cos\left(\frac{\sqrt{3}k_x a}{2}\right)\cos\left(\frac{k_y a}{2}\right) + 4\cos^2\left(\frac{k_y a}{2}\right)\right]^{1/2} \tag{1}$$

where

E_{2D} is the two dimensional energy dispersion relation for a single graphene sheet;

a is the lattice parameter[3];

k_x and k_y are the components of the reciprocal unit vector;

γ is the overlap integral.

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